Office of Environmental Laboratory Certification

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The *UPDATE*

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What's New…

We have bid farewell to a longtime colleague. Amy Bennett has decided to pursue some personal goals. We all will miss her, but we celebrate with her in her new adventure. If Amy was your contact in the past, please feel free to contact our office and ask for an LCO. Anyone of us will be happy to help you with any questions.

Matrix Spikes???

As a general rule, Clean Water Act procedures require matrix spikes at a frequency of 10% (one sample out of every ten analyzed or one per batch, whichever is more frequent) and Solid/Hazardous Waste procedures require matrix spikes at a frequency of 5% (one sample out of every twenty analyzed or one per batch, whichever is more frequent). So what defines a batch? A group of samples analyzed using the same equipment and reagents and analyzed within the same time frame. This is typically one analysis sequence and lasts no longer than 12 analytical hours. For organic analyses requiring extractions, a batch is processed at the same time using the same reagents. Typically this will be no more than 16-24 hours for continuous liquid-liquid extractors or a 12-hour shift for manual separatory funnel extractions. Herbicide extraction and esterification procedures may take longer.

How do you know which sample to spike? It is best to choose a sample that has a concentration of the analyte in question. The analyst may know this based on past data. The sample should not have too high of a concentration of the analyte already present as the matrix spike will go beyond the range of the calibration. If you do not have prior knowledge as to the sample's concentration, choose a sample and spike it. If the concentration of the spiked sample was too high to fall within the calibration range, another sample must be selected, spiked, and analyzed. It is best, if at all possible, to determine which sample to spike after analyzing a few samples. For example, for ammonia analyses, once the analyst has analyzed ten samples, he or she can choose one of the samples analyzed as the best candidate for spiking. Keep in mind that the same sample must not always be chosen. Over time, all samples should be spiked to demonstrate freedom from interferences in each of the sites received by the laboratory.

How do you know how much spike solution to add? The spiked sample should read toward the middle to upper portion of the calibration range. The analyst needs to spike the sample at approximately the same amount found in the sample unless the sample has no analyte present. For instance, if the calibration curve is from 1ppm to 50ppm, the analyst would try to select a sample that when spiked will yield between 20 and 40ppm. This is not a hard and fast rule, rather a guidance on the ideal situation. See the example below:

If the unspiked sample result (USR) is: 10ppm
The calibration range for the analysis is: 1ppm to 50ppm

The analyst will want to spike the sample with 25ppm of the analyte. 25ppm is an easy value to prepare. The analyst should always choose values that are easy to prepare to ensure that preparation does not adversely affect the spike recovery. The spiked sample should read around 35ppm.

How do you prepare the spiked sample? The spiked sample must be prepared by diluting the sample as little as possible. This means that the analyst should not dilute the sample by any significant amount. In other words, it is best to add 1mL of the stock spiking solution to 99mL of the sample. This results in only a 1% dilution and does not need to be accounted for in the final calculation. There are two reasons for avoiding sample dilution. First, you are trying to determine whether or not the sample has any interferences. If you dilute the sample by 10%, 25%, 50%, or more, you may be diluting out the interference that you are trying to identify. This defeats the purpose of running the spike. Secondly, if the sample is undiluted, calculating the spike recovery is easy. Having to account for dilutions makes the calculation more difficult and easier to make errors. See the following example:

Correct:

- 100mL of the sample are used for the analysis
- 99mL of sample are used for the spiked sample and 1mL of the spiking solution is added to the 99mL of sample. This results in no additional dilution of the sample.

Incorrect:

- 100mL of sample are used for the sample analysis and 50mL sample and 50mL of spiking solution are used to prepare the spiked sample, the matrix of the sample is diluted by a factor of 2. This could negatively affect the determination of matrix effects.

How do you calculate percent recovery? For matrix spikes, the calculation is different than it is for standards. We will only discuss matrix spikes in this article to minimize confusion. The formula to use is:

$$\frac{SSR - USR}{SA} \times 100 = \text{Percent Recovery}$$

Where: SSR = Spiked Sample Result
USR = Unspiked Sample Result
SA = Spike Added

Here's an example:

The sample volume used for analysis was 100mL
The spike was prepared by adding 1mL of 1000ppm stock spiking solution to 100mL of sample
The sample read 10ppm (USR)
The spiked sample read 17ppm (SSR)
The spike added was 10ppm

The percent recovery would be:

$$\frac{17ppm - 10ppm}{10ppm} \times 100 = 70\% \text{ recovery}$$

In the past, the following calculation was used:

$$\frac{SSR}{USR + SA} \times 100 = \% \text{ recovery}$$

This formula does not reflect the actual recovery because the percent recovery is biased by the high values contributed by the sample. The percent recovery using this formula is:

17 / (10 + 10) = 85% recovery

If the sample must obtain 80-120% recovery to be acceptable, the first calculation would indicate that the procedure did not yield 80% recovery of the spike amount added to the sample. It is important to determine the actual recovery of the spike added. Another example that is more clearly identified is below:

The sample read 100ppm
The spiked sample read 120ppm
The spike added was 50ppm

[(SSR – SR) / SA] x 100 = [(120 – 100) / 50] x 100 = [20 / 50] x 100 = 40% recovery

[SSR / (USR + SA)] x 100 = [120 / (100 + 50)] x 100 = [120 / 150] x 100 = 80% recovery

It is easy in this example to see that the analyst only recovered 20ppm of the 50ppm added to the sample. It would not be correct to use the second calculation which makes it appear that the analyst actually recovered 80% of the spike amount added.

As mentioned before, it is also important to add enough spiking solution to ensure that a recovery can be obtained. If the analyst only adds 1ppm to a sample that reads 100ppm, the analyst will probably not get any actual recovery of the spike. The methods employed by environmental labs are not generally precise enough to differentiate between 100ppm and 101ppm. So if:

The USR is 100ppm
The SA is 1ppm
The SSR is 100ppm

[(SSR – USR) / SA] x 100 = [(100 – 100) / 1] x 100 = [0 / 1] x 100 = 0% recovery

and

[SSR / (USR + SA)] x 100 = [100 / (100 +1)] x 100 = [100 / 101] x 100 = 99% recovery

As you can see, the analyst did not recover any of the spike added to the sample. Depending on the calculation used, the analyst could rely on faulty information to determine whether or not the matrix spike passed the criteria.

For this reason, it is of utmost importance that the analyst use the correct recovery calculation for all matrix spike recoveries.

[(SSR – USR) / SA] x 100 = % recovery.

Updated Website…

The Office website continues to be updated routinely to include new information and more forms. Please visit the site at www.scdhec.net/labcert and give us your comments and suggestions. The site is enabling laboratories to speed the application process by having downloadable forms such as the application. *If you are in need of the WP and/or WS study parameter sheet, you can now get these right off the website.*

Just a Reminder….

In order to maintain certification in South Carolina, acceptable PT sample results for the laboratory must be received by December 31st each year for the Water Supply (WS) and/or Water Pollution (WP) studies. In the past, upon review of the PT data, a decertification letter would be sent to laboratories that did not report acceptable results. The laboratory *had* 15 days from the receipt of the letter to submit acceptable results to avoid decertitication. This is no longer the case. **The laboratory will be decertified upon receipt of this decertification letter. For this reason it is critical that the laboratories have acceptable PT results submitted to this Office no later than December 31st. Please ensure that your laboratory ID number is on the PT report and that the correct method(s) for which the laboratory is certified for is reported.**

Applying for Certification…

When applying for certification, please be sure to include all requested information and ensure that the information is properly labeled. This will help speed the application process. The website offers application checklists which list all of the required documentation to be submitted. For organic analyses, the Method Detection Limit (MDL) and Initial Demonstration of Capability (IDOC) studies must be completed with all required information. This includes the sample preparation and clean-up METHOD NUMBERS used. The summary sheets submitted must include only one instrument. A summary sheet of MDLs for all instruments in use in the laboratory is not acceptable.

Please submit only the application fee when applying. The laboratory will be invoiced for any applicable parameter fees. If the laboratory submits a check for more or less than $125.00, the application cannot be processed until a check for exactly $125.00 is received. Please contact this Office with specific questions regarding the application process.

Proficiency Study (PT) Results…

To ensure that your laboratory is properly credited with the PT results submitted, the report submitted by the PT Provider to this Office **must include the LABORATORY'S EPA AND STATE ID NUMBERS**. This Office receives results from almost 700 laboratories. Most laboratories submit 4 or more PT study results. With this volume of data being received and processed, if the laboratory's ID numbers do not appear on the reports, it is possible that the laboratory may not be properly credited. **This also applies to the method number reported. The laboratory will likely receive a decertification letter if the incorrect method number is reported.** Please review your results when you get them from the PT Provider to ensure that the ID numbers and method numbers appear on your results as they appear on your SC certificate. **For out-of-state laboratories, your EPA ID number must appear on the results.** Ensuring that this information is on the reports will avoid confusion and possible decertification. **Even if the laboratory reports these numbers to the PT Provider, they do not always appear on the PT Provider's report sent to this Office. Please ensure that your copy of the results from the PT Provider includes all required information.**

The *Update*…

The *Update* can be downloaded from the internet at www.scdhec.net/labcert and selecting "Past and Current Issues of the *Update*". Due to many resource limitations in the Office, only one publication per certified laboratory may be mailed. If there are other people in your organization who would benefit from receiving this publication, they can download a copy from the internet. We are also studying the possibility of sending the *Update* via e-mail. If this is of interest to you or other persons in your organization, please e-mail Connie Turner at turnercp@dhec.state.sc.us.

On-Line Turbidimeters…

Calibration – Quarterly using the Manufacturer's Instructions

Calibration of the on-line turbidimeter for both combined filter effluent monitoring and individual filter turbidity must be performed at least quarterly according to the manufacturer's instructions. It is very important to follow the manufacturer's instructions for calibration; this will help to ensure that calibration verification results are within the specified limits.

Maintenance must also be performed quarterly or more frequently if problems are noted. The lamp should be changed on an annual basis. An SOP must be available documenting the procedure used by the laboratory for calibration as well as maintenance of the on-line turbidimeters.

A bench-top turbidimeter must not be used for calibrating the on-line turbidimeter. This can introduce error into the readings.

After calibration, the calibration must be verified by using the calibration verification procedure listed below.

If a contractor is used to perform the quarterly and/or annual maintenance and/or calibration, the laboratory must receive and maintain documentation from that contractor. This documentation must describe the process used to perform the service, instrument readings obtained, name of the person performing the service, the company that the contractor for whom the contractor works with a telephone number and address, and the date of service.

Calibration Verification

Calibration verification of on-line turbidimeters must be performed weekly for both combined filter effluent monitoring and individual filter turbidity.

Calibration verification must be performed using primary or secondary standards at 0.5 NTU. Results of the calibration verification standard must be within $\pm$ 10% of the true value unless the manufacturer of the on-line turbidimeter has more stringent requirements. Records must be maintained for the weekly calibration verifications and if results do not meet the $\pm$ 10% criteria, records must indicate that the on-line turbidimeter has been recalibrated and is capable of reading the calibration verification standard within $\pm$ 10% of the true value.

Records must be maintained by the laboratory for all calibrations and calibration verifications performed on the on-line turbidimeters along with any maintenance performed. The minimum information required is the date and time, analyst's initials, standard true value, standard reading, indication if maintenance is performed, and the standard source (i.e. formazin, StablCal™, ICE-PIC™, etc.) for the primary and secondary standards.

***Note:** When using the ICE-PIC™ for calibration verification of the 1720C Hach turbidimeters, the value of the ICE-PIC™ may vary between different 1720C turbidimeters. It is very important to follow the procedure specified by Hach to assign a unique value to the ICE-PIC™ for each 1720C turbidimeter. Records must document the values assigned to the ICE-PIC™ for each 1720C turbidimeter.

Definitions:

Calibration: A procedure, which checks or adjusts an instrument's accuracy by comparison with a defined standard or reference.

Calibration Verification: A procedure used to check whether or not the calibration of the instrument is within certain limits.

Primary standard: A standard used to calibrate the instrument response with respect to analyte concentration. Formazin, StablCal, and AMCO-AEPA-1 are considered primary standards by the EPA for turbidity.

Secondary standard: A standard that the manufacturer (or an independent testing organization) has certified will give instrument calibration results equivalent (within certain limits) to the results obtained when the instrument is calibrated with primary standard i.e., formazin. A secondary standard is used for daily or weekly calibration verification and is monitored periodically for deterioration using one of the primary standards. Please follow the manufacturer's instructions for determining the true value for the ICE-PIC for each individual 1620C instrument the laboratory uses.

WEASC Laboratory Committee – Frequently Asked Questions

The Laboratory Committee of the WEASC is compiling an on-going list of frequently asked questions regarding calculating and reporting laboratory results for SCDHEC regulatory compliance (DMRs, MORs, etc).

To compile this list we are requesting that written questions, with specific examples, be sent to the Laboratory Committee. The Committee will attempt to answer each question by utilizing SCDHEC, USEPA and private sector professionals.

Results of these findings will be reported by the Committee on an on-going basis to the WEASC membership.

Send questions with specific examples in writing to:

Ms. Ann Sims
WCRSA
660 Mauldin Road
Greenville, SC 29607
Phone: (864) 299-4040 x320
FAX: (864) 299-4059
e-mail: asims@wcrsa.org

Questions can be sent anonymously, however, we encourage a name and telephone number in case clarification of the question is required.

EPA Methods 1631E and 1669…

Sampling Requirements for EPA Method 1631E

- All equipment, bottles, tubing, etc must be pre-cleaned and tested to ensure freedom from contamination prior to use.
- Field blanks must be collected from the same site at the same time with each set of samples up to 10 samples.
- Samplers must use gloves that are non-powdered.
- A peristaltic pump must be used for filtering samples for dissolved mercury. The sample must be filtered through an encased filter.
- Samples must be collected as grab samples and the bottles filled with no headspace remaining.
- The sampler must not breathe on the sample if he/she has mercury amalgom fillings.
- All sampling equipment must be non-metallic.
- Sample bottles must be Teflon or glass.

- To meet all equipment and blank criteria, the sampling personnel must obtain equipment, bottles, tubing, filters, etc from the laboratory that will analyze the samples so that the equipment can be properly cleaned and tested prior to use.

Laboratory Obligations for Sampling Equipment for EPA Method 1631E

- The laboratory must perform reagent blank tests to ensure that the reagents are free of contamination.
- The laboratory must test all equipment, tubing, bottles, collection containers, etc prior to use to ensure freedom from contamination.
- The laboratory must clean all equipment according to EPA Method 1631E prior to testing the equipment using equipment blanks and releasing the equipment for use.
- The laboratory must prepare and analyze the following blanks after performing the appropriate cleaning procedures as described in EPA Method 1631E: bottle blanks, sampler check blanks, and reagent blanks.
- The laboratory must provide the field sampling team with contamination free reagent grade water in separate carboys for each sampling site.
- Track the sampling equipment and bottles, if reusable, to ensure that contamination is minimized and properly traced if present.

Laboratory Required QC for EPA Method 1631E

This section deals with specific QC requirements for EPA Method 1631E that are specified in and may be unique to Method 1631E. The published EPA Method must be consulted for all QC requirements regarding this method.

- The laboratory must calibrate using calibration factors or weighted linear regression curves. This is different from the techniques used in the traditional methods. The use of calibration factors is highly recommended.
- The EPA has published a document to assist laboratories in creating appropriate analysis areas for low-level metals analysis, including mercury. The title of the document is “Guidance on Establishing Trace Metal Clean Rooms in Existing Facilities” and can be obtained from the EPA website at www.epa.gov or by contacting Water Resource Center, Mail Code RC-4100, 401 M Street SW, Washington DC 20460. There are several examples of clean areas that have been set up in existing laboratories.
- Samples must be preserved within 48 hours of sampling.
- QC must be defined per the analytical batch. An analytical batch is a set of samples oxidized with the same batch of reagents and analyzed during the same 12 hour shift. The batch must be accompanied by three bubbler or system blanks, an OPR, and a QCS.
- For bubbler type analyzers, three bubbler blanks must be analyzed with each analytical batch. The mean bubbler blank is subtracted from the sample results if it is less than 25pg with a standard deviation of less than 10pg. If the bubbler blank fails, corrective action must be taken and samples reanalyzed.
- For flow injection systems, system blanks must be analyzed. The system blank must contain less than 0.50ng/L.
- Three system blanks, for flow injection systems, must be run during calibration and with each analytical batch. The mean result for the three system blanks must be less than 0.5ng/L mercury with a standard deviation
  (n-1) of less than 0.1ng/L.
- The reagent blank must be less than 20pg.
- Field blanks must be analyzed immediately prior to samples from that site.
- On-Going Precision and Recovery (OPR) must be performed at the beginning of the analytical batch and at the end or at the end of the 12 hour shift.

- OPR samples are followed by bubbler blanks. QCS samples are standards prepared from a separate source from the calibration standards.

Some Recent Questions Regarding 1669 Sampling:

Q: Can sampling procedures other than those described in EPA Method 1631E be used?

A: Yes. Alternate procedures may be used as long as the bottle blanks demonstrate freedom from contamination. EPA Method 1631E addresses specific procedures for preparing and analyzing bottle blanks.

Q: Does each bottle need to be tested prior to use or can a representative set per lot be tested?

A: No. Each bottle does not need to be tested as long as the lot is tested. If the box or bag contains more than twenty samples, one bottle for each 20 bottles must be tested. For example, if a box contains 12 bottles, only one must be checked. If a box contains 21 bottles, 2 bottles must be tested. If 29 bottles are in a box, 2 bottles need to be tested. If 45 bottles are in a box, 3 bottles must be tested.

Q: What type of bottles can be used for sample collection?

A: Sample bottles must either be glass or Teflon™.

Q: Can samples be preserved in the field?

A: It is not recommended. The note on page 14 of EPA Method 1631E states "Because of the potential for contamination, it is recommended that filtration and preservation of samples be performed in the clean room in the laboratory. However, if circumstances prevent overnight shipment of samples, samples should be filtered and preserved in a designated clean area in the field in accordance with the procedures given in Method 1669 (Reference 16.9). If filtered in the field, samples ideally should be filtered into the sample bottle."

Q: Are samples collected as grab or continuous composite?

A: If a permit specifies continuous composite sample, it has an error and must be corrected. Samples must be collected as grabs rather than continuous composites. Please contact the permit writer for questions regarding sites having multiple outfalls.

Trace Metals and PQLs…

Can a laboratory pre-concentrate samples to meet PQLs?

The only provision for pre-concentrating samples is for drinking water. The Drinking Water regulations allow up to a 4x concentration to meet required limits. The purpose of the PQLs in wastewater (NPDES compliance) and the rationale for the PQLs listed are to meet the lowest limits possible with the current technology. This means that if your instrumentation is not sensitive enough to analyze a standard at the PQL listed, it should not be used. Another, more sensitive technique, must be used. This may result in contracting with another laboratory. The PQL is defined as the lowest standard analyzed on the initial calibration curve. A list of PQLs used for NPDES compliance can be found on the webpage at www.scdhec.net/labcert.

Organic Analysis Update

Is it acceptable for a standard to be dropped from the initial calibration curve?

It is acceptable for a laboratory to drop a standard from the initial calibration curve if there is an assignable reason such as poor purge, bad injection, etc. It is not acceptable to drop multiple points from the curve in order to meet the calibration acceptance criteria, and it is not acceptable to drop only certain compounds from select standards unless it is the high or low point limited by compound sensitivity or the linear range of the specific compound.

The laboratory must meet all QC requirements in the approved EPA Methods and follow the actions prescribed in those methods when QC fails. The laboratory must also use and maintain a system of documentation that makes all sample data traceable to daily and initial calibration data and all relevant QC.

Are surrogates required in all pesticide calibration standards for EPA Method 8081A?

The toxaphene and technical chlordane mixtures do not need to contain the surrogates, if the pesticide, toxaphene, and chlordane calibration is performed at the same time under the same operating conditions and is perceived as a single calibration event. Surrogate calibration can be performed as part of the pesticide calibration mixture. Therefore the calibration date for the pesticides, surrogates, toxaphene, and chlordane will be the same and the calibration data should be traceable through the data system reports.

The data system reports for the calibration verification standards and samples where toxaphene and chlordane are quantitated would not document the surrogates since the surrogates are quantitated using the pesticide calibration data. The data system reports for the calibration verification standards for the pesticides would reflect the quantitation of the surrogates whereby the percent difference or % drift can be reviewed and traced to the initial calibration data.

Must toxaphene and technical chlordane be analyzed as part of the calibration verification procedure for EPA Method 8081A?

If toxaphene and technical chlordane are analytes of concern, both must be analyzed as part of the calibration verification procedure. When analyzing for pesticides using EPA Method 8081A, calibration verification must include the pesticide standards, toxaphene and technical chlordane to ensure calibration stability as multi-component pesticides. Calibration verification should be performed using high and low concentration mixtures of single component analytes and multicomponent analytes for calibration verification. If the calibration verification standard response for any analyte is not within $\pm$ 15% of the true value then the standard(s) must be reinjected. If it fails the 15% criteria again the multi-level calibration must be performed again using the pesticides, toxaphene, and technical chlordane as a single calibration event. The calibration verification standards must also be used to evaluate retention time stability. Each injection of a standard during the 12-hour analytical shift or with each calibration verification standard injected must be checked against the established retention time windows. If any fall outside the retention time window the GC system is out of control. If the problem cannot be corrected, a new initial calibration must be performed.

The method specifically requires samples to be bracketed by passing QC. A CV that passes the 15% drift/difference criteria along with retention time stability must be obtained before analyzing samples. If not, the data must not be used for regulatory reporting in South Carolina.

Can single-point quantitation be performed for toxaphene and technical chordane by EPA Method 8081A?

It is strongly recommended that multi-point calibration be performed and used for quantation of toxaphene and chlordane. Single-point quantitation can be used for toxaphene as long as the multi-point calibration

linearity has been established. When using single-point calibration, it is required that the sample size be adjusted so that the major toxaphene peaks are 10-70% of full scale deflection. Then inject a toxaphene standard that is estimated to be within ± 10 ng of the sample amount. Because this can be very time consuming most laboratories use multilevel calibration and quantitation. Quantitate toxaphene using total area of the toxaphene or using 4 to 6 major peaks. The laboratory must develop a SOP for quantitation of toxaphene to ensure consistency for calculating sample results.

Single-point quantitation can also be used for technical chlordane as long as the multi-point calibration linearity has been established. When using single point the amount of technical chlordane standard injected must produce a chromatogram in which the major peaks are approximately the same size as those in the sample chromatogram. Because this can be very time consuming, most laboratories use multilevel calibration and quantitation. Quantitate technical chlordane using total area or using 4 to 5 major peaks. There are several options given in Method 8081A for quantitation of technical chlordane. The standard chromatograms must have enough resolution to clearly show the toxaphene and technical chlordane peaks and pattern. It is important that the analyst review and print chromatograms that clearly show the major peaks and baseline in these mixtures. Identification is made based on the presence and ratios of the major peaks in the samples.

Must the PCB calibration be performed as a single calibration event when using EPA Method 8082?

A separate calibration event (separate from the pesticides) is required for the PCBs, because of the multi-component nature for PCB quantitation and problems with potential identification and possible interferences. The surrogate(s) must be included in the PCB standards and used for quantitation of the surrogates in calibration verification standards and samples where PCBs are analytes of interest. The laboratory can use multi-level calibration for all of the PCBs or according to SW-846 EPA Method 8082 multi-level calibration data can be performed for 1016/1260 documenting calibration linearity requirements and single point calibration can be used for the other PCBs. The laboratory must be consistent in their analysis procedures for PCBs.

How is calibration verification performed for the PCBs?

The calibration verification standards for the PCBs (when using the 4 to 6 major peak approach for quantitation) must be evaluated by reviewing each peak's response against the 15% (drift or difference) criteria required for calibration verification. The practice of averaging the peaks responses to meet the 15% criteria is not an acceptable practice, since it can provide greater uncertainty in the sample results. Quantitation is then performed by using the average concentration of the selected PCB peaks to provide the final results for the sample.

The standard chromatograms must have enough resolution to clearly show the PCB peaks and pattern. It is important that the analyst review and print peaks that clearly show the major peaks and baseline in these mixtures. Identification is made based on the presence and ratios of the major peaks in the samples.

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